TOTAL KJELDAHL NITROGEN by ASTM D3590-02 (REAPPROVED 2006)

Facility Name:_____VELAP ID_____

Assessor Name:_____Analyst Name:_____Inspection Date_____

Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Records Examined: SOP Number/ Revision/ Date _____ Analyst:_____ Sample ID: _____ Date of Sample Preparation:_____ Date of Analysis:_____					
Were samples preserved with sulfuric acid to pH<2 and held for not longer than 28 days at 4°C?	7.2				
Method A Manual Digestion/Distillation					
Was spectrophotometer or colorimeter used at 425 nm with a spectral band path of not more than ±20 nm and light path of 1 cm or longer?	10.3				
Was distillation apparatus cleaned with the steam of a solution of 1 part water and 1 part sodium hydroxide-thiosulfate solution (500 g NaOH + 25 g $Na_2S_2O_3$/ 1 L H_2O)?	12.1				
Were 0.1N sulfuric acid solutions prepared by diluting 3 mL concentrated H_2SO_4 to 1 L in water?	11.12				
Were 0.1N sulfuric solutions diluted to 0.02N and standardized against 0.0200 N solution of 140°C-oven-dried Na_2CO_3?	11.12				
Were digestion solutions prepared by diluting 267 g K_2SO_4 + 400 mL concentrated H_2SO_4 + 50 mL mercuric sulfate solution to 2 L with water?	11.13				
Macro System					
Were portions of digestion solution added to sample aliquots, and the sample aliquots digested until SO_3 fumes were given off and solution turned colorless or pale yellow?	12.2.2				
Method B Semiautomated Colorimetric					
Was a digestion solution containing potassium sulfate , sulfuric acid, and mercuric sulfate added to well-mixed samples at a rate of 5 mL/ 20 or 25 mL?	21.1.1				
Notes/Comments:					

TOTAL KJELDAHL NITROGEN by ASTM D3590-02 (REAPPROVED 2006)					
Relevant Aspect of Standards	**Method Reference**	**Y**	**N**	**N/A**	**Comments**
Was digester block preheated to 200°C prior to the placing of samples on it?	21.1.1				
Were digestion containers first heated at 200°C for 1 hour?	21.1.2				
Were digester block temperatures next raised to 380°C, and digestion containers heated for about 1.5 hours?	21.1.2				
Were digested samples cooled to about room temperature after digestion?	21.1.3				
Were sampler wash receptacles washed with sulfuric acid wash solution prior to sample analysis?	21.2.3				
Were reagents allowed to pump through analytical device for about 5 minutes and a stable baseline obtained prior to sample analysis?	21.2.4				
Were portions of sodium hydroxide-thiosulfate solution added to digestates without mixing until distillation apparatus was assembled?	12.2.3				
Were condenser tips immersed into the 2% Boric Acid solution?	12.2.4				
Micro System					
Were portions of digestion solution added to sample aliquots, and the sample aliquots digested until SO_3 fumes were given off and solution turned colorless or pale yellow?	12.3.1				
Were sample aliquots digested for an additional 30 minutes after SO_3 fume evolution and pale yellow/colorless change?	12.3.1				
Were portions of sodium hydroxide-thiosulfate solution added to digestates without mixing until distillation apparatus was assembled?	12.3.2				
Were condenser tips immersed into the 2% Boric Acid solution?	12.3.3				
Were portions of distillate set aside to analyze by colorimetry if nitrogen concentrations were below 1 mg/L?	12.3.4				
Were distillates titrated with 0.02 N H_2SO_4 using a blank containing the same volume of water and boric acid as the end-point match or a pH of 6.2?	12.4.1				
When colorimetry was used, were distillates + Nessler's reagent (100 g HgI_2 + 70 g KI + 160 g NaOH/1 L water) mixed, settled for about 20 minutes, and read on a 425 nm colorimeter using 1 cm cells?	12.4.2				
Notes/Comments:					